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METHODS FOR CHEMICAL ANALYSIS OF FOUNDRY NICKEL

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 4 *February* 1968

Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF FOUNDRY NICKEL

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Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF FOUNDRY NICKEL

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 30 November 1967, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 For the correct determination of the chemical composition of foundry nickel generally used in ferrous foundries, need was felt to prescribe standard methods for its analysis. Due consideration has been given in the preparation of this standard to the facilities available in the country for such analysis.

0.3 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard prescribes methods which cover the complete analysis of foundry nickel, the general composition of which is specified in IS : 2391-1963†.

2. SAMPLING

2.1 Samples shall be drawn and prepared in accordance with the procedure laid down for the purpose of chemical analysis under IS : 1472 (Part II)-1962‡.

3. QUALITY OF REAGENTS

3.1 Unless otherwise specified, pure chemicals and distilled water (*see* IS : 1070-1960§) shall be employed in the tests.

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*Rules for rounding off numerical values (*revised*).

†Specification for foundry nickel.

‡Methods of sampling ferro-alloys, Part II.

§Specification for water, distilled quality (*revised*).

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

4. GENERAL

4.1 Use of Filter Papers — In some of the methods given in this standard, only relative numbers of Whatman filter papers have been given since they are commonly used. However, any other brand of filter paper of corresponding porosity may also be used.

5. DETERMINATION OF SILICON BY THE PERCHLORIC ACID METHOD

5.1 Outline of the Method — The sample is dissolved in concentrated nitric acid and silicic acid is dehydrated with perchloric acid. Silicon is determined by loss in weight after volatilization with the hydrofluoric acid in presence of sulphuric acid.

5.2 Reagents

5.2.1 *Concentrated Nitric Acid* — sp gr 1·42 (conforming to IS : 264-1950*).

5.2.2 *Perchloric Acid* — 70 percent.

5.2.3 *Dilute Hydrochloric Acid* — 5 : 95 (*v/v*).

5.2.4 *Dilute Sulphuric Acid* — 1 : 5 (*v/v*).

5.2.5 *Hydrofluoric Acid* — 40 percent.

5.2.6 *Potassium Bisulphate* — solid.

5.3 Procedure

5.3.1 Weigh accurately 3 g of the sample, transfer to a 400-ml beaker, add 25 ml of hot concentrated nitric acid, cover the beaker with watch-glass and keep it on the hot-plate. If necessary more acid should be added to bring the metal in solution. When action ceases, add 25 ml of perchloric acid. Evaporate the solution to dense white fumes. Then boil the solution gently for 10 minutes at such a rate that the bulk of the perchloric acid vapour condenses on the beaker wall and is retained. Allow the solution to cool, add 100 ml of warm water and stir until the perchlorates have dissolved. Filter the residue through Whatman filter paper No. 40 and wash thoroughly with hot dilute hydrochloric acid and then with hot water, till all perchloric acid is washed off. Reserve the filtrate.

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*Specification for nitric acid.

5.3.2 Transfer the paper and the precipitate to a platinum crucible. Char the paper carefully, cover the crucible and ignite at 1 050°C to a constant weight. Cool in a desiccator and weigh.

5.3.3 Moisten the ignited silica with a few drops of dilute sulphuric acid, add 10 ml of hydrofluoric acid, evaporate to dryness and continue heating until all the sulphuric acid fumes are completely eliminated. Complete the ignition in the muffle at 1 050°C. The loss in weight represents silica.

5.3.4 Fuse the residue in the platinum crucible with 2 g of potassium bisulphate, cool and extract with dilute sulphuric acid and mix with the filtrate reserved under **5.3.1** and make up to 500 ml in a volumetric flask. Reserve the solution for determinations of nickel, cobalt and iron.

5.3.5 Carry out a blank determination following the same procedure and using the same amounts of all reagents, but without the sample.

5.4 Calculation

$$\text{Silicon, percent} = \frac{(A - B) \times 46{\cdot}72}{C}$$

where

A = weight in g of silica obtained in **5.3.3**,
B = weight in g of silica obtained from the blank determination, and
C = weight in g of the sample taken.

6. DETERMINATION OF NICKEL BY THE DIMETHYLGLYOXIME (GRAVIMETRIC) METHOD

6.1 Outline of the Method — Nickel is precipitated by dimethylglyoxime from the solution reserved under **5.3.4** and weighed as nickel dimethylglyoxime.

6.2 Reagents

6.2.1 *Citric (Tartaric) Acid Solution* — 250 g/l.

6.2.2 *Concentrated Ammonium Hydroxide* — 20 percent.

6.2.3 *Dimethylglyoxime Solution* — one percent (*w*/*v*) in rectified spirit. Filter the solution before use.

6.3 Procedure

6.3.1 Transfer exactly an aliquot of 25 ml from the solution reserved under **5.3.4** to a beaker and add 20 ml of citric (tartaric) acid solution. Neutralize with concentrated ammonium hydroxide until a slight odour is detected. Dilute the solution to about 500 ml and heat to 60° to 80°C.

6.3.2 Add 40 ml of the dimethylglyoxime solution for each 0·1 g of the nickel present. Add 5 to 10 ml in excess. Render the solution slightly alkaline with concentrated ammonium hydroxide and digest at 60°C for about 30 minutes. Stir vigorously and allow to cool to room temperature slowly while stirring occasionally. Filter the precipitate on a weighed sintered glass crucible (G-3; 30 to 50 ml capacity) and wash it with warm water. Keep the filtrate overnight to make sure that nickel has been completely precipitated.

6.3.3 Dry the sintered glass crucible alongwith the precipitate at 130°C to constant weight. Cool in a desiccator and weigh as nickel dimethylglyoxime.

6.4 Calculation

$$\text{Nickel, percent} = \frac{A \times 20{\cdot}32}{B}$$

where

A = weight in g of nickel dimethylglyoxime, and

B = weight in g of the sample represented by the aliquot taken.

7. DETERMINATION OF IRON BY THE DICHROMATE (VOLUMETRIC) METHOD

7.1 Outline of the Method — Iron is precipitated as hydroxide from the solution preserved under **5.3.4**. To the boiling solution, stannous chloride is added in slight excess to reduce iron. The unused stannous chloride is destroyed by mercuric chloride and the reduced iron titrated with standard dichromate solution using sodium diphenylamine sulphonate as indicator.

7.2 Reagents

7.2.1 *Ammonium Chloride* — solid.

7.2.2 *Concentrated Ammonium Hydroxide* — 20 percent.

7.2.3 *Dilute Ammonium Hydroxide* — 5 : 95 (*v/v*).

7.2.4 *Dilute Hydrochloric Acid* — 1 : 1 and 5 : 95 (*v/v*).

7.2.5 *Stannous Chloride Solution* ($SnCl_2,2H_2O$) — Dissolve by heating 60 g of pure stannous chloride in a mixture of 400 ml of concentrated hydrochloric acid, and 600 ml of water. Cool, add a few pieces of granulated tin and preserve the solution in air-tight amber-coloured bottle to prevent oxidation.

7.2.6 *Mercuric Chloride Solution* — saturated in water.

7.2.7 *Sulphuric-Phosphoric Acid Mixture* — Add slowly 150 ml of concentrated sulphuric acid (sp gr 1·84) to 700 ml of water with continuous stirring. Add to this 150 ml of phosphoric acid (85 percent). Destroy any oxidizable impurities by adding potassium permanganate solulion (0·1 N) drop by drop until the pink colour of permanganate persists and cool the solution.

7.2.8 *Sodium Diphenylamine Sulphonate Indicator Solution* — Dissolve 0·2 g of the reagent in 100 ml of hot water.

7.2.9 *Standard Potassium Dichromate Solution* — 0·02 N. Dry pure potassium dichromate (AR) in an oven at 105° to 110°C for one-and-a-half hours. Cool in a desiccator and transfer exactly 0·980 6 g to a one-litre volumetric flask carefully through a funnel using water at room temperature. Wash the funnel thoroughly and dissolve the salt by shaking until the solution is complete. Make up to the mark and mix well.

7.3 Procedure

7.3.1 Transfer exactly 100 ml of the solution reserved for iron determination under **5.3.4** to a 500-ml beaker and heat. Add 4 to 5 g of ammonium chloride. Add concentrated ammonium hydroxide until strongly ammoniacal and boil for one minute. Filter the precipitate and wash with dilute ammonium hydroxide. Dissolve the precipitate in hot dilute hydrochloric acid (1 : 1), reprecipitate with concentrated ammonium hydroxide as before and filter the precipitate. Dissolve the precipitate in 20 ml of dilute hydrochloric acid (1 : 1) collecting the solution in a 500-ml conical flask and washing the paper with hot dilute hydrochloric acid (5 : 95).

7.3.2 Heat the solution to boiling and to the boiling solution, add stannous chloride solution, drop by drop, with continuous stirring until the solution just becomes colourless. Add 2 drops in excess of stannous chloride solution and wash down the sides with a little water. Cool the flask rapidly under running tap water until the contents have cooled down to room temperature.

7.3.3 Add excess of mercuric chloride solution (about 5 ml) and shake. At this stage, a silky white precipitate appear. If it fails to appear or if a blackish precipitate appears, reject the test. If the precipitate is too much, add further 5 ml of mercuric chloride solution. Keep for about 2 to 5 minutes and add 15 ml of sulphuric-phosphoric acid mixture, and dilute to about 250 ml, washing down the sides of the flask. Add 3 to 4 drops of sodium diphenylamine sulphonate indicator solution and titrate slowly with standard potassium dichromate solution stirring continuously until a drop of the potassium dichromate solution produces a reddish-violet colouration.

7.3.4 Carry out a blank determination following the same procedure and using the same amounts of all reagents, but without the sample.

7.4 Calculation

$$\text{Iron, percent} = \frac{(A - B)\,C \times 5{\cdot}585}{D}$$

where

A = volume in ml of the standard dichromate solution required for the sample,

B = volume in ml of the standard dichromate solution required for the blank,

C = normality of the standard dichromate solution, and

D = weight in g of the sample represented by the aliquot taken.

8. DETERMINATION OF COBALT BY THE NITROSO-R-SALT (PHOTOMETRIC) METHOD

8.1 Outline of the Method — Cobalt in the filtrate obtained from **5.3.4**, buffered with sodium acetate, forms an orange-coloured complex with nitroso-R-salt. Photometric measurement of the complex is made at approximately 520 millimicrons (mμm).

8.2 Reagents

8.2.1 *Dilute Hydrochloric Acid* — 1 : 1 (*v/v*).

8.2.2 *Nitroso-R-Salt Solution* — one percent (*w/v*). Dissolve 0·10 g of nitroso-R-salt in water, filter and dilute to 100 ml. Do not keep the solution for more than a week.

8.2.3 *Sodium Acetate* — solid.

8.2.4 *Concentrated Hydrochloric Acid* — sp gr 1·16 (conforming to IS : 265-1962*).

8.2.5 *Standard Cobalt Solution* (1 ml = 0·1 mg of Co) — Dissolve 0·67 g of pure cobalt ammonium sulphate (AR) [$CoSO_4.(NH_4)_2SO_4, 6H_2O$] in water and dilute to one litre.

8.3 Procedure

8.3.1 Transfer a suitable aliquot from the filtrate preserved under **5.3.4** so that it contains between 0·005 and 0·1 mg of cobalt.

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*Specification for hydrochloric acid (*revised*).

Evaporate almost to dryness; dissolve the residue in 10 ml of water containing 0·5 ml each of dilute hydrochloric acid and nitroso-R-salt solution. Add 2 g of sodium acetate. The *p*H of the solution at this point should be 5·5. Boil for a minute, cool and add 1·5 ml of concentrated hydrochloric acid. Boil further for a minute, cool and dilute to 50 ml in a volumetric flask.

8.3.2 Transfer a suitable portion of the solution prepared under **8.3.1** to an absorption cell and take the photometric reading using light filter at 520 millimicrons (mμm).

8.3.3 Take a series of standard solutions of cobalt and a reagent blank. Using the same quantities of reagents carry through all stages as described under **8.3.1** and **8.3.2**, and record the photometric readings of all the standard solutions along with the blank. Construct a calibration curve by plotting the photometric readings of standard solutions against the quantities of cobalt.

8.3.4 Convert the photometric reading of the sample to milligrams of cobalt by means of the calibration curve and calculate as under **8.4**.

8.4 Calculation

$$\text{Cobalt, percent} = \frac{A}{B} \times \frac{1}{10}$$

where

A = weight in mg of cobalt found in the aliquot of the solution, and

B = weight in g of the sample represented by the aliquot taken.

9. DETERMINATION OF TOTAL CARBON BY THE DIRECT COMBUSTION (GRAVIMETRIC) METHOD

9.1 Outline of the Method — The sample is heated to combustion at a high temperature (1 200° to 1 300°C) in a stream of pure oxygen and the carbon is converted to carbon dioxide. The carbon dioxide, carried by oxygen, is passed through and absorbed by soda asbestos in a tared bulb, the increase in weight of which is proportional to the quantity of carbon dioxide formed.

9.2 Reagents

9.2.1 *Oxygen* — 99·5 percent purity free from carbonaceous matter.

9.2.2 *Anhydrous Magnesium Perchlorate* — [$Mg(Cl0_4)_2$]

9.2.3 *Flux* — lead dioxide, analytical reagent grade.

9.2.4 *Manganese Dioxide* — If a suitable chemical active grade is not available, it may be prepared as follows:

a) To prepare about 50 g of manganese dioxide, dissolve in a 4-litre beaker 200 g of manganous sulphate ($MnSO_4$, $4H_2O$) in 2·5 litres of water. After making this solution clearly ammoniacal, add 1 litre of ammonium persulphate solution (225 g per litre) freshly prepared, bring to boiling point. Continue boiling for 10 minutes and add ammonia solution as frequently as is necessary to maintain the solution ammoniacal. Allow the precipitate to settle.

b) If the supernatant liquid is not clear, or if the precipitate does not settle quickly, add 50 to 100 ml of ammonium persulphate solution and boil again for 10 minutes keeping the solution constantly ammoniacal.

c) When precipitation appears to be complete, allow the manganese dioxide to settle completely, carefully siphon off the supernatant liquid and wash the precipitate by decantation with 3 or 4 litres of warm water in portions of 500 to 600 ml. Stir the manganese dioxide well in the water and allow to settle after each washing and before decantation. Finally wash twice in the same way with very dilute sulphuric acid.

d) In the meantime, prepare a 15-cm diameter funnel fitted with a 5-cm filter disc filled with a thin layer of purified asbestos pulp (in place of the latter, it is also permitted to use a porcelain funnel of the Buchner type).

e) After the last washing, transfer the manganese dioxide into the filter and wash with warm water until it is free from sulphate ions. Then place this on a porcelain dish and dry it in an oven at 105°C.

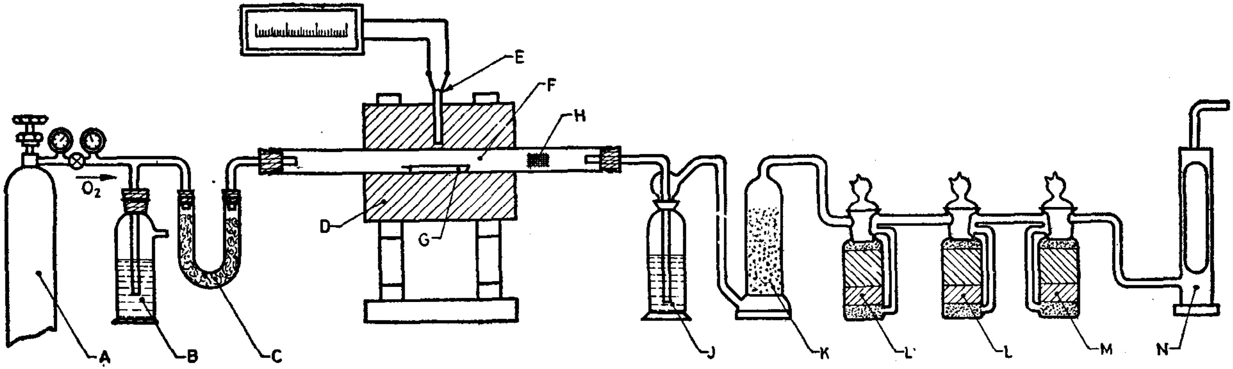
f) Grind the manganese dioxide in a mortar so that it will pass through a sieve with apertures of 0·8 mm and dry again completely at 105°C.

9.2.5 *Soda Asbestos* — Asbestos impregnated with sodium hydroxide, in granules of about 2 mm diameter. Avoid contact with air.

9.3 Apparatus — The apparatus consists of a source of oxygen and the unit for purifying it, the furnace with the combustion tube, the purification train and the carbon dioxide absorption system. These different parts, which are joined together with connecting tubes forming an air-tight seal, are shown in Fig. 1.

9.4 Procedure

9.4.1 After having verified the pressure tightness of the apparatus and the efficiency of the oxygen purification and having reached the required temperature within the range of 1 200° to 1 300°C in the



A	Source of oxygen (**9.2.1**) with pressure regulating valve.
B	Mercury valve.
C	Unit for drying and purifying the oxygen, containing anhydrous magnesium perchlorate (**9.2.2**) and soda asbestos (**9.2.5**) separated by glass wool (diameter of tubes 25 mm, height 100 mm approximately) connected by tubing.
D	Wire-wound or resistor rod furnace, made of carborundum or metal and capable of a temperature for combustion up to 1 000°C.
E	Thermocouple for measuring the temperature. The tip of the thermocouple protected by a sheath, is placed near the external surface of the combustion tube. The relation between the internal tube temperature and the pyrometer reading should be established.
F	Combustion tube made of refractory material which is not porous at the test temperature, internal diameter 20 to 30 mm and at least 650 mm long so that the ends of the tube remain cold during combustion.
G	Boat made of refractory material (length 80 to 100 mm, width 12 to 14 mm, depth 8 to 9 mm), calcined in a stream of oxygen at the temperature of use.
H	Plug of calcined asbestos wool or quartz wool to filter the gas.
J	Washing bulb containing manganese dioxide (*see* **9.2.4**).
K	Drying bulb containing anhydrous magnesium perchlorate (*see* **9.2.2**).
L	Weighed absorption bulbs for absorbing the carbon dioxide, containing soda asbestos (**9.2.5**) and a certain amount of anhydrous magnesium perchlorate (**9.2.2**) to trap the water which forms during absorption of the carbon dioxide. The total weight of each absorption bulb ready for use (*see* Fig. 2) should not exceed 100 g.
M	Unweighed absorption bulb facing the opposite way to *L*.
N	Flowmeter.

FIG. 1 COMBUSTION APPARATUS FOR DETERMINATION OF CARBON BY GRAVIMETRIC METHOD

combustion zone of the furnace, allow oxygen to pass through the apparatus for 10 to 15 minutes at the rate of 300 to 500 ml of gas per minute depending on the diameter of the tube used. Then disconnect the absorption bulbs and weigh them at the ambient temperature and replace them in position.

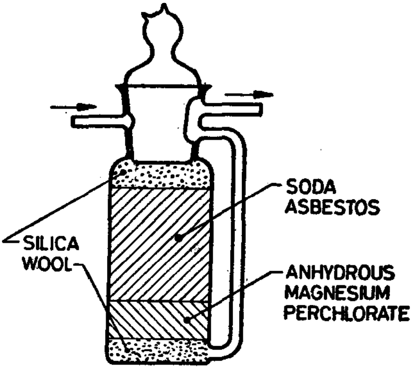


Fig. 2 Absorption Bulb

9.4.2 Place one g of an accurately weighed sample in the form of very short chips having a thickness of a few tenths of a millimetre, in a boat. If necessary, add to the sample 1 to 2 g of flux (**9.2.3**).

9.4.3 Open the combustion tube at the end where the oxygen enters and using a rigid nickel wire, place the boat and the sample in the middle of heated zone of the tube. Quickly close the tube and, after about 1 minute, pass through it a stream of oxygen at the rate of 300 to 500 ml per minute, depending on the diameter of the tube used. After about 1 minute, then the combustion has ended, continue to pass oxygen through for another 15 to 20 minutes so that the carbon dioxide is completely removed from the combustion tube and the purifying bulbs.

9.4.4 Cut off the stream of oxygen, close the taps of the weighed absorption bulbs and remove the porcelain boat from the combustion tube. It is necessary to verify that the combustion has been efficiently completed by examining the fused weight in the boat removed from the furnace.

9.4.5 Disconnect the absorption bulbs and weigh them at ambient temperature. The increase in weight represents the carbon dioxide absorbed.

9.4.6 Carry out the blank test by the combustion of the boat in the presence of the quantity of flux used in the analysis, and carefully determine the weight of the absorption bulb.

9.5 Calculation

$$\text{Carbon, percent} = \frac{A - B}{C} \times 27{\cdot}3$$

where

A = weight in g of carbon dioxide obtained under **9.4.5**,
B = weight in g obtained from the blank, and
C = weight in g of the sample taken.

10. DETERMINATION OF SULPHUR BY THE BARIUM SULPHATE (GRAVIMETRIC) METHOD

10.1 Outline of the Method — Sulphur in the sample is oxidized to sulphate and determined gravimetrically as barium sulphate.

10.2 Reagents

10.2.1 *Sodium Chlorate* — solid.

10.2.2 *Concentrated Nitric Acid — see* **5.2.1**.

10.2.3 *Concentrated Hydrochloric Acid — see* **8.2.4**.

10.2.4 *Hydroxylamine Hydrochloride* — solid.

10.2.5 *Barium Chloride Solution* — 20 percent (*w/v*).

10.2.6 *Barium Chloride Wash Solution* — 0·2 percent (*w/v*). Add 10 ml of concentrated hydrochloric acid (*see* **8.2.4**) to 10 ml of the solution prepared under **10.2.5** and dilute to 1 : 1.

10.3 Procedure

10.3.1 Transfer 5 g of an accurately weighed sample to a 800-ml beaker. Add 2 to 3 g of sodium chlorate and 150 ml of concentrated nitric acid when vigorous reaction starts. When reaction subsides, evaporate the solution till it is syrupy. Allow the solution to cool and then add 50 ml of concentrated hydrochloric acid and evaporate the solution to dryness. Bake the residue on a hot-plate until the salts assume uniform colour. Repeat the baking process after adding 50 ml of concentrated hydrochloric acid.

10.3.2 Cool the baked mass and then moisten with 7 ml of concentrated hydrochloric acid. Heat the mixture at 60° to 70°C until iron oxides have been converted to chlorides guarding against any appreciable loss of concentrated hydrochloric acid by evaporation. Add 150 ml of water and boil the solution until the salts have been dissolved. Filter the solution through a filter paper in a beaker and wash with hot water.

10.3.3 Dilute the filtrate and washings to 350 ml and heat to boiling. Add 1 to 2 g of hydroxylamine hydrochloride, add 25 ml of barium chloride solution slowly with constant stirring and allow the precipitate to digest on a water-bath for one hour. Remove from the water-bath and allow to stand overnight. Filter off the precipitate using filter paper No. 42 and wash the paper twice with barium chloride wash solution and finally with hot water till it is free from chloride.

10.3.4 Transfer the filter and precipitate to a platinum or porcelain crucible, dry and ignite at a low temperature until free of carbonaceous matter, then finally at about 800°C. Cool in a desiccator and weigh the residue as barium sulphate.

10.3.5 Make a blank determination, following the same procedure and using the same amounts of all reagents, but without the sample.

10.4 Calculation

$$\text{Sulphur, percent} = \frac{(A - B) \times 13{\cdot}74}{C}$$

where

A = weight in g of barium sulphate obtained under **10.3.4**,

B = weight in g of barium sulphate in the blank determination, and

C = weight in g of the sample taken.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

QUANTITY	UNIT	SYMBOL
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

QUANTITY	UNIT	SYMBOL
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	1 N = 1 $kg.m/s^2$
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2

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Western	Novelty Chambers, Grant Road	BOMBAY 400007	37 97 29
Eastern	5 Chowringhee Approach	CALCUTTA 700072	27 50 90
Southern	CIT Campus	MADRAS 600113	41 24 42
Northern	B69, Phase VII	S. A. S. NAGAR (MOHALI) 160051	–

Branch Offices:

'Pushpak', Nurmohamed Shaikh Marg, Khanpur	AHMADABAD 380001	2 03 91
'F' Block, Unity Bldg, Narasimharaja Square	BANGALORE 560002	22 48 05
Gangotri Complex, Bhadbhada Road, T.T. Nagar	BHOPAL 462003	6 27 16
22E Kalpana Area	BHUBANESHWAR 751014	5 36 27
5-8-56C L. N. Gupta Marg	HYDERABAD 500001	22 10 83
R 14 Yudhister Marg, C Scheme	JAIPUR 302005	6 98 32
117/418 B Sarvodaya Nagar	KANPUR 208005	4 72 92
Patliputra Industrial Estate	PATNA 800013	6 28 08
Hantex Bldg (2nd Floor), Rly Station Road	TRIVANDRUM 695001	32 27

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